Apparatus for impregnating thin-layer plates

Adsorbent layers impregnated with hydrophobic materials are commonly used to separate the members of relatively non-polar classes of compounds, such as glycerides and fatty acid esters. Suggested methods for impregnating include immersion of the plate in the impregnating solution[1,2], spraying[3], and allowing the plate to stand in the solution—as when developing the chromatogram[4,5]. The immersion method may present handling difficulties and requires great care to avoid disturbing loosely-bound adsorbent layers. Spraying may also disturb the adsorbent or result in uneven deposition of the impregnant. The development method obviates these difficulties but is more time-consuming and entails the possibility that due to frontal analysis the impregnated portion may not extent to the solvent front.

With the device here described (Fig. 1) an immersion method is used, but the impregnating solution is allowed to flow onto the plate, and off again, at a controlled rate. In this way the above-mentioned difficulties are avoided.

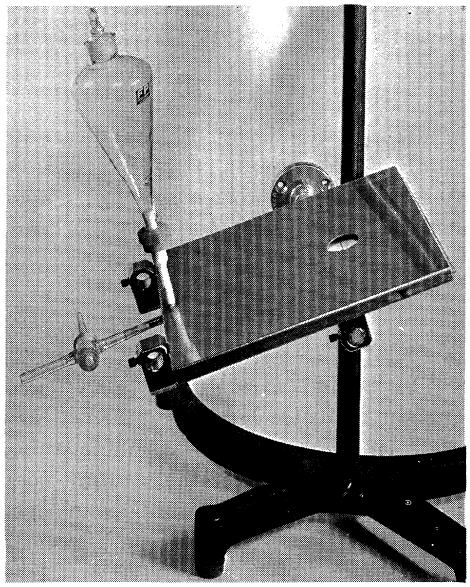

Fig. 1. Apparatus for impregnating thin-layer plates.

Method of operation

As shown in Fig. 1, the apparatus consists of an impregnating chamber resting on a Flexaframe* support which can be adjusted at various angles to the horizontal. The impregnating solution is placed in a separatory funnel connected to an inlet near the bottom of the chamber; the thin-layer plate rests against the ledge shown in Fig. 2, detail E. The funnel stopcock is opened and the solution allowed to flow into the chamber until it reaches the proper height as observed through the window

* Mention of trade or company names does not constitute endorsement by the Department of Agriculture over others not named.

(Fig. 2, D) in the top of the chamber. The funnel stopcock is then closed and the outlet stopcock opened.

The permissible flow-rate for the impregnating solution depends on the cohesiveness of the adsorbent layer and the angle of the chamber. With a 250 μ layer of silica gel containing 5 % $CaSO_4$ as binder, and with the chamber set at an angle of approxi-

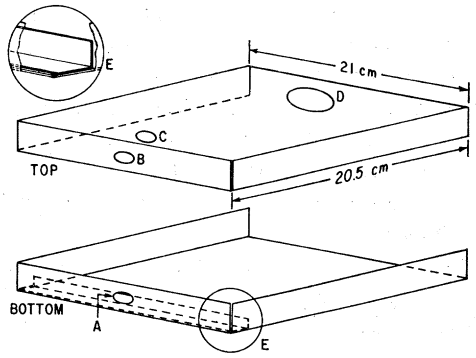

Fig. 2. Exploded view of impregnating chamber. Not drawn to scale; see text for complete dimensions.

mately eight degrees from the horizontal, 180 ml of solution is enough to impregnate the adsorbent for a distance of 15 cm in 5 min. Smaller amounts of solution may be used if more time is taken for the impregnation or if the chamber is set at a smaller angle. In the latter case, however, care must be taken to prevent the solution from flowing under the plate and out the open end of the chamber.

Construction

The chamber is made from two sheets of 20-gauge 18-8 stainless steel, as shown in Fig. 2. Each sheet is cut so as to have a 20.5 by 21 cm central portion surrounded on three sides by 1 cm wide tabs. Five millimeter diameter holes are punched out at A, B, and C, and a 25 mm diameter hole at D. Hole C, which is for the inlet tube, must be fairly close to the edge of the sheet so that the impregnating solution does not fall directly on the adsorbent; hole D has its center 4.5 cm from the opposite edge.

After the holes are made the tabs are bent in such a way that the top sheet can fit like a lid over the bottom. Fifteen millimeter lengths of 5 mm O.D. stainless steel tubing are then flanged at one end, fitted into holes B and C, and sealed in place with 50-50 solder (lead-tin) and stainless steel flux. An L-shaped strip of stainless steel with one arm 10 mm wide and the other 5 mm wide is spot-welded to the bottom sheet (see detail E). Top and bottom sheets are then put together and soldered along the joints.

A Fisher and Porter Company* 125 ml separatory funnel with Ultramax valve is fitted to the inlet tube. This funnel has an extension tube rigid enough to hold it erect without further support. A stopcock is fitted to the outlet. All plastic parts which come in contact with the impregnating solution should be Teflon.

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* 18 Warminster Road, Warminster, Pa. 18974, U.S.A.

The chamber is supported on two parallel Flexaframe rods. These are connected by a third rod which extend through a clamp support on a stand and then through two Flexaframe feet arranged with their flat faces touching. The latter serve as a handle for adjusting the angle of the chamber.

Eastern Utilization Research and Development Division, JOSEPH H. SCHWARTZ
Agricultural Research Service, U.S. Department of Agriculture, ADOLPH PADE
600 East Mermaid Lane, Philadelphia, Pa. 19118 (U.S.A.)

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